## G. Life Prediction of Diesel Engine Components

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## **Objectives**

- Generate database and characterize damage mechanisms of candidate advanced ceramics and intermetallic alloys.
- Apply and verify probabilistic life prediction and component design and verification for advanced diesel engine components.

### **Approach**

- Evaluate the dynamic fatigue and rotary bending fatigue performance of candidate silicon nitride ceramics and titanium aluminide (TiAl) alloys at elevated temperatures in air before and after long-term exposure to simulated engine environments and before and after engine field tests.
- Characterize the evolution and role of damage mechanisms and changes in microstructure that are linked to the long-term mechanical performance and reliability of ceramics and intermetallic alloys.
- Use life prediction codes to predict the probability of failure and reliability of complex-shaped components subjected to application conditions.

### **Accomplishments**

- Completed dynamic fatigue tests at temperatures of up to 1000°C in air for a commercial-grade silicon nitride, SN147-31N, purchased from Ceradyne, Inc.
- Completed flexural strength evaluation for an Ni<sub>3</sub>Al intermetallic alloy after 1000 h of exposure to exhaust gas environments.
- Completed a dynamic fatigue database for Kyocera SN235P silicon nitride fabricated with the same powder batch for Caterpillar exhaust valve blanks.
- Completed modification of a rotary bending fatigue test rig required for testing TiAl specimens at temperatures of up to 800°C in air.

#### **Future Direction**

- Develop a dynamic fatigue database for a commercial-grade silicon nitride, SN147-31E, purchased from Ceradyne, Inc.
- Develop a dynamic fatigue database for flexure test bend bars machined from Kyocera SN235 exhaust valve blanks to verify mechanical performance.
- Develop a rotary bending fatigue database for TiAl alloys after oil immersion testing at temperatures of up to 800°C in air.
- Evaluate the mechanical properties of prototype SN235P silicon nitride exhaust valves after bench rig and preliminary engine tests and verify the probabilistic component life prediction.

## **Introduction**

Advanced ceramics and intermetallic alloys have potential for extensive use in applications in advanced diesel engine systems because of their superior thermomechanical properties at elevated temperatures. Implementation of components fabricated from these advanced materials would lead to significant improvements in engine efficiency and longterm durability and to reduction of NO<sub>x</sub> and CO exhaust emissions, as required in the 21st Century Truck Program. Interest in these materials has focused primarily on research in characterization and design methodology development (life prediction) for advanced silicon nitride ceramics and TiAl alloys to enable the manufacture of consistent, reliable complex-shaped components for diesel engine applications. The valid prediction of mechanical reliability and service life is a prerequisite for successful implementation of these advanced materials as internal combustion engine components.

Three primary goals of this research project contribute toward successful implementation:

 generation of a mechanical engineering database of results of performance tests of candidate materials from ambient to high temperatures, conducted before and after exposure to simulated engine environments;

- microstructural characterization of failure phenomena in these advanced materials and in components fabricated from them;
- application and verification of probabilistic life prediction methods using diesel engine components as test cases.

For all three stages, results will be provided to both the material suppliers and component end-users to help them refine and optimize processing parameters. Doing so will help them achieve consistent mechanical reliability and validate the probabilistic design and life prediction of engine components made from these advanced materials.

## **Approach**

All silicon nitride test bend bars were longitudinally or transversely machined, according to ASTM standard C1161,<sup>1</sup> from production billets purchased from material suppliers. Flexure testing was conducted in ambient air in four-point bending using 20–40-mm, α-SiC, semi-articulating fixtures at temperatures ranging from 20 to 1000°C and at a stressing rate of 30 MPa/s and 0.003 MPa/s. The 30 MPa/s test condition was chosen to evaluate the inert characteristic strength as a function of temperature, while the 0.003 MPa/s test condition was chosen to measure the change in slow crack growth (SCG) susceptibility at elevated temperatures. Pneumatic actuators were programmed to

produce the desired loading rate (and corresponding stressing rate) via a personal computer. Load was continuously measured as a function of time, and flexure strength was calculated using ASTM C1161. The accumulated strength data were then further analyzed. The strengths for each test set were fit to a two-parameter Weibull distribution using the program CERAMIC,<sup>2</sup> which uses maximum likelihood estimation advocated in ASTM C1239.3 Reported results are uncensored because fractography analysis was not conducted in detail to identify strength-limiting flaws for all of the bend bars tested. Following dynamic fatigue tests, both optical and scanning electron microscopy (SEM) analysis were carried out on fracture surfaces and polished crosssections of selected bend bars to characterize the fracture and degradation mechanisms. Xray analysis was also carried out to evaluate the possible phase changes resulting from oxidation during dynamic fatigue testing or after long-term exposure to simulated engine environments, which could possibly cause degradation in mechanical performance and reliability.

#### Results

# Dynamic Fatigue Response of Advanced Silicon Nitride Ceramics

At present there are a limited number of material suppliers that could provide consistent and reliable material resources and supporting efforts to the end users. The lack of committed materials suppliers could hamper the smooth implementation of silicon nitride ceramic components for advanced diesel engine applications. Thus it is important to extend the database generation efforts to other potential candidate silicon nitride ceramics, especially those manufactured by domestic material suppliers. Studies of dynamic fatigue behavior in four-point bending for a commercial-grade silicon nitride (i.e., SN147-31N, Ceradyne Advanced Ceramic

Operation, Inc., CA) were carried out in FY 2003. The database generated will be compared with those previously generated for SN235 and SN235P silicon nitride (Kyocera Industrial Ceramics Corp., WA). Note that both SN235 and SN235P were down-selected for prototype exhaust valve evaluation because of their excellent resistance to SCG and diesel engine environments. The database will also be provided to Caterpillar for probabilistic life prediction for exhaust valve components.

Dynamic fatigue test results at 20 and 850°C showed that the longitudinally machined SN147-31N exhibited inert characteristic strength values between those obtained for SN235 and for SN235P (as shown in Table 1). In addition, the SN147-31N exhibited Weibull moduli comparable to the values obtained for SN235 and SN235P under the same test conditions. In addition, the strength versus stressing rate curves showed that the SN147-31N exhibited a fatigue exponent of 47 and 131 at 20 and 850°C, respectively (Figure 1). The increase in fatigue exponent with increased test temperature could indicate the onset of SCG and/or creep deformation processes, evident by the permanent curvature observed for those SN147-31N bend bars fractured after testing at 850°C and 0.003 MPa/s.

The effect of machining orientation on the mechanical response of SN147-31N silicon nitride was also evaluated in FY 2003. Mechanical tests for transversely machined bend bars would be more representative of strength obtained for the ceramic valves, which would be transversely machined in the actual component production lines. Results of dynamic fatigue tests at 20 and 850°C showed that the transversely machined SN147-31N exhibited inert characteristic strengths about 14–19% lower than those obtained for the longitudinally machined specimens (Table 1). A similar reduction in strength of transversely machined samples was also previously reported for the cases of SN235 and SN235P silicon nitride. Also, SN147-31N revealed

**Table 1.** Summary of uncensored Weibull and strength distributions for Ceradyne SN147-31N silicon nitride and Kyocera SN235P specimens. Data of previously tested transversely machined

Kyocera SN235/SN235P are used for reference.

Material	No. tested	Stressing rate (MPa/s)	°C	Uncensored Weibull modulus	± 95% Uncensored Weibull modulus	Uncensored characteristic strength (MPa)	± 95% Uncensored characteristic strength (MPa)
SN235	15	30	20	23.8	15.4, 33.9	901	879, 923
SN235P	15	30	20	38.1	24.0 55.8	666	656, 676
SN147- 31N*	15	30	20	21.73	14.07. 31.09	836	814, 858
SN147- 31N*	15	0.003	20	20.70	13.38, 29.67	694	675, 713
SN147- 31N**	15	30	20	13.76	8.96, 19.59	677	649, 705
SN235P- CP***	30	30	20	11.94	8.58, 15.94	820	794, 847
SN235P- CP***	30	0.003	20	14.56	10.93, 18.64	741	721, 761
SN235	15	30	850	26.7	18.0, 36.7	777	760, 793
SN235P	15	30	850	19.3	12.5, 27.6	631	612, 649
SN147- 31N*	15	30	850	20.35	13.58, 28.20	777	755, 799
SN147- 31N**	15	30	850	18.26	11.67, 26.47	639	619, 659
SN235	14	0.003	850	18.5	11.8, 26.8	744	720, 767
SN235P	15	0.003	850	18.2	11.5, 26.8	594	575, 612
SN147- 31N*	15	0.003	850	16.19	10.57, 23.02	732	706, 757
SN147- 31N**	15	0.003	850	19.95	12.83, 28.63	620	602, 638
SN235P- CP***	30	30	850	20.56	15.16, 26.87	684	671, 697
SN235P- CP***	30	0.003	850	19.44	14.49, 25.01	621	608, 633

\*Denotes SN147-31N specimens were longitudinally machined, \*\*denotes SN147-31N specimens were machined transversely, and \*\*\*denotes SN235P-CP specimens were longitudinally machined from co-processed billets of exhaust valve blanks.

strength values comparable to those of SN235P under the same machining and test conditions. In addition, there were minor differences in Weibull moduli between longitudinally and transversely machined SN147-31N specimens, suggestive of similar strength-limiting flaws governing the fracture of specimens. The obtained Weibull moduli were also comparable to those reported for SN235 and SN235P under the same test conditions.

On the other hand, the strength versus stressing rate curves showed that the SN147-

31N exhibited high fatigue exponents (N ~ 132 and 343) at 850°C, independent of the machining orientation (Figure 2). However, a permanent curvature was observed for those SN147-31N bend bars after testing at 850°C and 0.003 MPa/s, indicating onset of SCG and/or creep processes, presumably as a result of the softening of the secondary glassy phase. The contribution of the softening of secondary phase(s) to SCG/creep processes has also been reported for Honeywell GS44 silicon nitride. EM examination on the fracture surface of

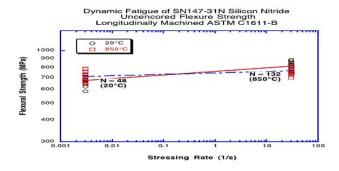


Figure 1. Strength vs. stressing rate curves of SN147-31N silicon nitride, longitudinally machined.

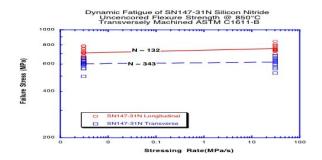


Figure 2. Strength vs. stressing rate curves of SN147-31N silicon nitride, transversely machined. Data from specimens longitudinally machined are used for reference.

selected SN147-31N samples tested at 850°C and 0.003 MPa/s showed the presence of glassy ligaments on silicon nitride grains, indicating the softening of the secondary phase at test temperature (as shown in Figure 3). The addition of oxygen in the secondary phase along the crack paths will further decrease the softening temperature and viscosity, facilitating the time-dependent degradation processes. Therefore, dynamic fatigue results suggest that the application limit of SN147-31N silicon nitride will be < 800°C in air.

Studies of dynamic fatigue for Kyocera SN235P silicon nitride, manufactured with the same powder batch employed for the exhaust valve blanks, were also carried out in FY 2003. The objective of this study was to verify the mechanical performance of coprocessed SN235P (designated as SN235P-CP)

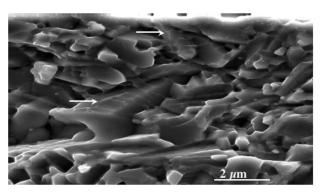


Figure 3. SEM fracture surface of SN147-31N specimen tested at 850°C and 0.003 MPa/s. Note that glassy ligaments observed on elongated grains indicated the softening of secondary phase at temperature.

for Caterpillar exhaust valve application. Note that the SN235P-CP MOR bend bars were machined using the revised ASTM C116 standard with a 600-grit surface finish. Results of dynamic fatigue tests at 20°C showed that the SN235P-CP silicon nitride exhibited inert characteristic strengths that were about 9% lower and 23% higher than those previously obtained for SN235 and SN235P silicon nitride, respectively (Table 1). The higher inert characteristic strength of the SN235P-CP could be attributed to the finer surface finish (600 grit) with respect to the 320-grit surface finish employed for the SN235P MOR bars tested previously. In addition, the SN235P-CP exhibited a lower Weibull modulus (m = 11.9) than the values obtained for SN235 (m = 23.8) and SN235P (m = 38.1), as shown in Table 1. The lower Weibull modulus obtained by SN235P-CP might result from the different strength-limiting flaw population present in the SN235P-CP with respect to that present in SN235P. Similar higher-strength response of SN235P-CP compared with SN235P was also observed at 850°C under the same stressing rate. On the other hand, the failure stress versus stressing rate curves at 20 and 850°C showed that the SN235P-CP exhibited high fatigue exponents (N ~ 111 and 93, respectively), indicating high resistance to the SCG process (Figure 4).

The high fatigue exponents obtained for SN235P-CP were consistent with the high N values previously obtained for SN235P as well as SN235. Therefore, dynamic fatigue results suggest that the SN235P-CP would meet the application criteria for exhaust valve components for advanced diesel engines.

# Mechanical Properties of Intermetallic Alloys

We also completed evaluating the mechanical properties of an Ni<sub>3</sub>Al intermetallic alloy, fabricated by the extrusion process by the University of Missouri, after 1000 h of exposure to a diesel engine exhaust gas environment at 850°C. The Ni<sub>3</sub>Al alloy has been considered as one of the candidate advanced intermetallic alloys for exhaust valve applications. The roomtemperature results showed that the Ni<sub>3</sub>Al

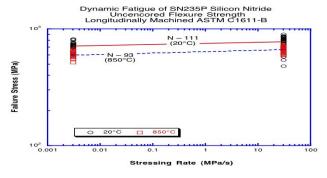


Figure 4. Failure stress vs. stressing rate curve of SN235P silicon nitride longitudinally machined and tested at 20 and 850°C in air. Note that the SN235P was machined from co-processed billets of exhaust valve blanks.

alloy exhibited no strength degradation after 1000 h of exposure, indicating excellent mechanical reliability in an exhaust gas environment (Figure 5). SEM examination of fracture surfaces of exposed bend bars showed the presence of  $Al_2O_3$  oxide scale (~10 µm thick) on the surface resulting from the oxidation of  $Ni_3Al$  (Figure 6); and the surface exhibited fractures that were intergranular in nature. Note that the as-

machined Ni<sub>3</sub>Al bend bars mostly failed as a result of the machining mark (flaw). In addition, energy-dispersive X-ray analysis showed no elements from exhaust gas were detected in the bulk region immediately underneath the oxide scale, suggesting good long-term chemical stability. A study of the effects of long-term oil immersion testing at 850°C in air will be planned to further confirm the mechanical reliability and chemical stability of Ni<sub>3</sub>Al alloy in diesel engine environments.

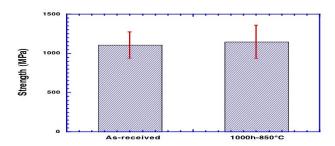
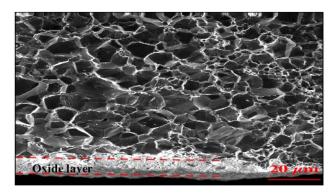


Figure 5. Flexural strength of Ni<sub>3</sub>Al alloy, asmachined and after 1000 h exposure in diesel exhaust gas environment.

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**Figure 6.** SEM fracture surface of Ni<sub>3</sub>Al bend bar after 1000 h exposure to an exhaust gas environment and tested at room temperature.

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## **Publications**

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